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(54) Title: METHOD FOR PREPARING CORN BRAN FOR EXTRACTION OF CORN FIBER OIL

(57) Abstract

The present invention relates to a method for preparing corn bran for solvent extraction of corn fiber oil from the corn bran, with the method involving preferably passing the corn bran through a pair of flaking steps to produce a flattened corn bran that can have an amount of corn fiber oil extracted therefrom.

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# METHOD FOR PREPARING CORN BRAN FOR EXTRACTION OF CORN FIBER OIL

## FIELD OF INVENTION

The present invention relates to a method for preparing corn bran so that corn fiber oil, found in the corn bran, can be more easily extracted, with the method preferably including a pair of flaking steps whereby the corn bran is flaked in a flaking roll.

## BACKGROUND OF INVENTION

Corn fiber oil, not to be confused with corn oil derived from corn meal, is known to contain nutritional constituents such as sterols, sterol esters, stanols, and stanol esters, as well as, triacylglycerols and other oils which are non-triacylglycerols. Importantly, the corn fiber oil contains an amount of phytosterol derivatives or plant sterols, which equal approximately 17% by weight of the total corn fiber oil, with the triacylglycerols and other non-triacylglycerols comprising the remaining constituents found in the corn fiber oil. The phytosterol derivatives are comprised of the sterols, stanols, and esters of the sterols and stanols already listed. More specifically, the corn fiber oil contains approximately 2% by weight of free sterols, 6% by weight of sterol ferulate esters, and 9% by weight of sterol fatty esters, also known as stanol esters, based on the weight of the corn fiber oil. The concentration of phytosterols in the corn fiber oil is higher than in other known oils. For example, rice bran oil contains an amount of sterol esters equal to about 1.5% by weight of the rice bran oil and wheat oil contains an amount of phytosterols equal to about 3.0% by weight of the wheat oil.

The phytosterol constituents are important because they can be used to reduce cholesterol levels in humans. It is known that ingestion by humans of phytosterols can help to lower cholesterol levels in the subject consuming the phytosterol, with the stanols, in particular, resulting in lower cholesterol levels in the subject. As such, the phytosterol constituents, or substances similar to phytosterols,

are used as cholesterol-reducing substrate ingredients found in both pharmaceutical and food based cholesterol-lowering products. Many of these compositions have proven to be effective in lowering cholesterol. Thus, inclusion of corn fiber oil in a person's diet can be beneficial to lowering cholesterol.

The corn fiber oil containing the phytosterols is found in corn bran and corn fiber. Corn bran is derived from dry milling kernels of corn that have not been steeped, so that the outer layers of the kernels are separated into what is known as the corn bran. The corn fiber is obtained through a wet milling process, which involves steeping the corn kernels followed by grinding and separating the outer layer of kernels from the remainder of the corn kernels. Both the corn bran and corn fiber are derived from the outer layers of the corn kernel. The corn bran and corn fiber both contain corn fiber oil and as such the corn bran and the corn fiber will be referred to throughout as corn bran. The corn bran is separated from the remainder of the corn kernel, including the corn meal, corn starch, and corn oil. The corn bran contains an amount of corn fiber oil equal to between about 3.0% and about 4.8% by weight of the total corn bran. Unfortunately, while the corn fiber oil is desirable for human health and has a variety of different uses, it suffers from being particularly difficult to extract from the corn bran. As such, it has not been known to commercially extract corn fiber oil from corn bran. The methods that are known are generally done on a lab scale and not on a scale designed to commercially harvest and produce corn fiber oil. The known methods involve grinding corn bran to a smaller particle size.

The corn fiber oil is difficult to extract because it has a low density and is tightly bound to the fiber comprising the corn bran. Past methods for extracting the corn fiber oil from the corn bran have included grinding the corn to an average of about a # 20 U.S. mesh particle size, with the grinding done, for example, in a pin mill. Once ground, the corn bran is then contacted with a solvent and some of the corn fiber oil is removed from the ground corn bran. Generally, no more than about 1.7% by weight of the corn fiber oil is extracted, meaning that if it is assumed that there is generally about 4.8% by weight of corn fiber oil in the corn bran, less than 36% by weight of the available corn fiber oil is extracted. To extract the corn fiber

oil, typically the ground corn bran is passed through a hexane extraction unit. Because of the fine particle size of the ground corn bran it tends to clog the extractor unit and consequently the hexane does not readily percolate through the corn bran to remove the corn fiber oil. Also, the small particle size and structure of the corn bran causes the extracted corn fiber oil to be clouded with debris from the ground corn bran. As can be seen, the prior methods for preparing the corn bran for extraction suffer from a number of disadvantages. In particular, it has been found that the prepared corn bran does not allow for extraction of a high percentage of the corn fiber oil, it readily clogs the extractor unit, and it results in a corn fiber oil contaminated with debris. Thus, it is desirable to have a preparation method that results in a corn bran that can have greater amounts of corn fiber oil extracted from the corn bran. It is also desired to have a method that results in a corn bran that does not clog a solvent extractor unit and that does not cause the corn fiber oil to be clouded with debris.

## SUMMARY

The present invention relates to a method for preparing corn bran for extraction of corn fiber oil from the corn bran. The present invention is advantageous because after treatment with the present method, the corn fiber oil can be more easily extracted from the corn bran so that a greater amount of corn fiber oil can be removed from the corn bran, as compared to other known methods. The corn bran treated according to the present method has a structure that does not clog an extractor unit. Also, the present method allows for a clean extraction of the corn fiber oil, meaning the corn fiber oil will be relatively free of debris. It should be pointed out that the present method will result in better extraction of corn fiber oil from the corn bran when all other factors are kept constant, in particular the condition of the corn bran prior to treatment. As such, the present method allows for better extraction of corn fiber oil than other known methods.

In the present method an amount of corn bran is obtained that preferably has a moisture content equal to between about 10% and about 25% by weight of the corn bran. A more preferred moisture level will be equal to about 15%

by weight of the corn bran. The corn bran is then passed through a first flaking roll device, to form a flaked corn bran. The flaking roll device is a piece of equipment that has a pair of adjacent cylindrical flaking rolls through which the corn bran is passed. After passage through the first flaking roll device, the flaked corn bran can then be passed through a second flaking roll device. The second flaking roll device can be a separate flaking roll or the same flaking roll used in the first step. Each passage of the corn bran through the flaking roll device is considered a flaking step, so that two passages through a flaking roll device is considered double flaking. It is most preferred in the present method to double flake the corn bran or to flake the corn bran at least twice. A variety of parameters, such as gap setting and speed differential, relating to a flaking roll device, comprised of a pair of flaking rolls, can be used so long as a flattened corn bran is produced. After flaking, it is preferred to dry the flaked corn bran so that the flaked corn bran has a moisture level equal to between about 2% and about 8% by weight of the corn bran. It has been found that drying prior to extraction increases the amount of corn fiber oil that can be extracted from the corn bran. The corn bran that has been double flaked or passed through two flaking roll devices is then ready for extraction of the corn fiber oil away from the corn bran.

To extract the corn fiber oil from the corn bran a variety of methods can be used; however, typically a solvent extraction method is used to remove the corn fiber oil from the corn bran. The solvent used should be a non-polar solvent. Regardless of how the corn bran is extracted, the present method should involve flaking the corn bran twice, although other methods can be used where the corn bran is flaked just once or flaked once and then ground to a smaller particle size. Thus, while double flaking is most preferred, it is necessary to flake the corn bran at least once. Also, the corn bran can be dried prior at any point prior to extraction, so that the corn bran can be dried prior to flaking or after flaking. Most preferably, the corn bran is dried after flaking and prior to extraction so that the corn bran has a moisture level ranging between about 2% and about 8% by weight of the corn bran.

The corn bran produced by the present invention is unique because it has a sheared construction, which appears to allow for easier percolation of solvent

through the corn bran and thus easier extraction of the corn fiber oil. Not only does the corn bran have a sheared construction, but it will have a moisture level equal to no more than 25% by weight of the corn bran. More preferably, the corn bran will have a moisture level, after drying, ranging between about 2% and about 8% by weight of the corn bran. The thickness of the corn bran after treatment will generally range between about 0.005 inches and about 0.015 inches. Also, the corn bran will have a particle size generally ranging between about #10 U.S. standard testing sieve average particle size and a #60 U.S. standard testing sieve average particle size. As such, it is believed the corn bran produced by the present method is unique and unlike any other known corn bran. It is believed that the unique characteristics of the corn bran allow for better extraction of the corn fiber oil from the corn bran.

## DETAILED DESCRIPTION

The present invention relates to a method for preparing corn bran for extraction of corn fiber oil from the corn bran. The corn bran, as discussed, includes both corn bran and corn fiber. The present method is especially advantageous because it preferably allows for the extraction of greater than about 2.0% by weight of the corn fiber oil in the corn bran or at least about 40% by weight of the available corn fiber oil in the corn bran. To achieve these removal characteristics, the method preferably includes at least twice passing an amount of corn bran through a flaking roll device so that the corn bran is at least double flaked. Flaking, or passing the corn bran through a flaking roll device, is a process whereby the corn bran is squeezed and compacted as it passes through the flaking roll device. Other steps can be included in the present method, but it is most preferred to flake the corn bran at least twice. It is preferred for the treated corn bran to have a moisture level equal to or less than 15% by weight of the corn bran prior to extracting the corn fiber oil from the corn bran. Upon exiting the flaking roll steps, the corn bran will be a sheared flattened corn bran.

The method is initiated by obtaining an amount of corn bran, which is also known as corn fiber, and, as mentioned, is derived from a corn kernel's hull. Specifically, the corn bran consists of the outer layers of the corn seed or kernel that are separated from the remainder of the seed during, for example, a wet milling process. The corn bran contains limited amounts of starch and protein, and an amount of corn fiber oil, which is generally believed to be present in amount equal to from about 2% to about 4.8% by weight of the corn bran. More typically, it is believed that the corn bran contains an amount of corn fiber oil equal to about 4.8% by weight of the corn bran. The corn bran will preferably be of a size that can pass through a #5 U.S. standard testing sieve, so that the corn bran has a #5 U.S. standard testing sieve average particle size. More preferably, the corn bran is smaller than a #10 U.S. standard testing sieve average particle size; however, other mesh particle sizes can be used. Preferably, the corn bran will contain an amount of water equal to no more than about 25% by weight of the corn bran; however, depending on how the corn bran is processed prior to flaking, it may contain a lesser amount of water. The corn bran prior to flaking typically has a density of about 10 lbs./ft$^3$, meaning that the corn bran has a relatively low density and that the oil will be tightly bound to the corn bran. Finally, it is preferred that the corn bran not be burnt or toasted, which can happen if the corn bran is dried prior to flaking at too high of a temperature for too long.

Once an amount of corn bran is obtained, it can be conditioned or cooked. The conditioning step is not required, but it can be included as part of the method. If the corn bran is conditioned, it is preferably done so that the corn bran is exposed to a temperature of greater than 90° F. In essence, conditioning is simply a process whereby the corn bran is cooked for a period of time; with that in mind, any of a variety of devices can be used to cook the corn bran.

Regardless of whether the corn bran is conditioned, it is preferred, but not required, to dry the corn bran prior to extraction. The corn bran can be dried prior to flaking so that it has a moisture content equal to or less than about 25% by weight of the corn bran. More preferably, the corn bran should have a moisture content equal to about 15% by weight of the corn bran. Regardless of whether the

corn bran is dried prior to flaking, it should be dried prior to extraction of the corn fiber oil away from the corn bran. It has been observed that the corn fiber oil is more readily extracted from dry corn bran than from wet corn bran. If the corn bran is dried, it should not be exposed to temperatures that will burn or toast the corn bran. As such, the corn bran can be dried in a variety of ways; however, it is preferred to dry the corn bran using low temperature drying of about 180° F. Keep in mind that the corn bran burns at a temperature of roughly 700° F, meaning any temperature below 700° F may be used.

The corn bran, whether it has been dried, conditioned, or merely separated from the remainder of the corn kernel, is passed into the flaking steps. The corn bran will preferably be flaked twice by passing the corn bran through the same flaking roll device twice or by passing the corn bran through two separate flaking roll devices, it does not matter. Double flaking or passing the corn bran through a pair of flaking steps is preferred in the present invention because it allows for the formation of a flattened corn bran that generally permits at least about 2% by weight of the corn fiber oil to be extracted from the corn bran or at least about 40% by weight of the available corn fiber oil to be extracted. The flaking steps help to achieve a greater extraction, because the corn bran, after passage through the flaking roll device, allows solvents to readily percolate through the corn bran and extract the corn fiber oil.

Double flaking involves taking an amount of corn bran and passing it through a first flaking roll device. To flake the corn bran, this is best accomplished by passing it through flaking roll equipment, wherein the equipment has a pair of flaking rolls set substantially adjacent to one another. The equipment will be referred to throughout as a flaking roll device and the parameters discussed herein relate to the two flaking rolls which makeup the flaking roll device, so that when the parameters are discussed they relate to the two adjacent flaking rolls which are part of the flaking roll device. Any of a variety of flaking roll devices can be used as long as the chosen device has two substantially adjacent flaking rolls through which the corn bran will pass. An example of an acceptable flaking roll is a device made by Bauer which is known as a Bauer flaking roll. The flaking rolls will preferably

have a gap setting of about 0.015 inches or smaller, as this will ensure that a thin corn bran flake is formed. Other gap settings can be used, dependent on the machinery used to flake the corn bran and the desired finished structure of the corn bran, with the available gap setting ranging between the flaking rolls touching or about 0 inches and about 0.015 inches. The gap setting is the distance between two flaking rolls which makeup the flaking roll device. The flaking rolls of the flaking roll device preferably rotate at different speeds, so that there is about a 10% speed differential between the rolls, referred to as a 10% differential. The speed differential causes shear forces, which influence the structure of the corn bran and allow for easier extraction of the corn fiber oil. While a 10% differential is preferred, the differential can range between about 1% and about 20%. Also, even though the differential is preferred, it is not required, as the rolls can rotate at the same speed. Generally, the rolls will rotate at a speed of greater than 280 revolutions per minute (rpms.), however, other speeds may be used. The speed of the rolls is primarily dependent upon how fast the corn bran can be flaked, meaning the speed with which the corn bran can be passed through the flaking roll without clogging the flaking roll. The size of the rolls can vary, with the chosen size primarily dependent upon the amount of corn bran it is desired to flake. The preferred size of the rolls generally ranges between about 12 inches and about 24 inches in diameter. The rolls may even have a diameter as large as 36 inches. Also, the rolls of the flaking roll device may be of the same general size or one roll may have a larger diameter than the other. Once the corn bran has passed through the flaking roll device, a flat corn bran flake is formed.

After passage through the first flaking roll device, the flaked corn bran is passed through a second flaking roll device to complete the double flaking process. Passage through the second flaking roll device can involve passage through the same flaking roll device that was used to initially flake the corn bran or a second independent flaking roll device, it does not matter. The flaking roll speed, the differential, and the construction of the second flaking roll device will typically be the same as the first flaking roll device.

The second flaking step is important because this allows for further flattening of the corn bran. The second step also further reduces thickness of the corn bran. Importantly, the additional flattening allows for a greater amount of the corn fiber oil to be extracted. The particle size of the corn bran after the second flaking step will generally range between about a #5 U.S. standard testing sieve average particle size and about a #60 U.S. standard testing sieve average particle size. The flattened corn bran will be sheared by the flaking roll steps, so that the corn bran has a sheared construction and will have a thickness ranging between about 0.005 inches and about 0.015 inches. The flattened corn bran allows a solvent for extracting the corn fiber oil to readily percolate through the corn bran and extract an amount of corn fiber oil from the corn bran. By forming the flattened corn bran, preferably at least 40% by weight of the available corn fiber oil can be extracted from the corn bran. Thus, the present method most preferably involves double flaking the corn bran.

Upon completion of flaking it is preferred to dry the flaked corn bran so that it will have a moisture level equal to between about 2% and about 8% by weight of the corn bran. More preferably, it is desired to dry the flaked corn bran so that it has a moisture content equal to between about 2% and about 4% by weight of the corn bran. It has been observed that drying the corn bran after flaking allows for extraction of a greater amount of corn fiber oil. The flaked corn bran can be dried at a variety of temperatures and times so long as the corn bran is not burnt or toasted. Preferably, the corn bran is dried at a temperature of no more than 180° F for a time period of about two (2) hours.

After the corn bran has been flaked, it can then be exposed to a solvent extraction system that removes an amount of the corn fiber oil from the flattened corn bran. Any of a variety of extraction methods can be used; however, it is preferred to use a hexane extraction method, as this is commercially acceptable and has proven effective in removing the corn fiber oil from the corn bran. The flattened corn bran will be exposed to a sufficient amount of hexane for a sufficient period of time to extract a portion of the corn fiber oil held by the corn bran. The method for extracting the corn fiber oil from the corn bran generally involves

exposing the corn bran to an amount of hexane for a period of time equal to between about 45 minutes and about 90 minutes. The amount of hexane added will be an amount sufficient to equal at least a 1:1 by weight ratio with the corn bran; but other amounts of hexane may be used. Other solvents besides hexane can be used, as long as the solvent is a non-polar solvent. Among the solvents that can be used to extract the corn fiber oil are supercritical $CO_2$, butane, isooctane, propane, hexane, acetone, isohexane, heptane, methylpentanes, and other non-polar solvents.

The hexane will separate the corn fiber oil from the corn bran to form a miscella, which is comprised of corn fiber oil and hexane. The hexane and corn fiber oil in the miscella must then be separated from one another so that the corn fiber oil can be isolated and used. Generally, this is done by heating the miscella to a temperature ranging between about 140° F and about 220° F. The miscella can be heated in a vacuum of between about 12 inches of mercury and about 15 inches of mercury, wherein the combination of the vacuum and heat will cause the hexane to vaporize away from the corn fiber oil. Any temperature can be used as long as the molecular structure of the corn fiber oil is not damaged.

The present method can be part of a continuous process or a batch process.

The following examples are for illustrative purposes only and are not meant to limit the claims in any way. Further, Examples 1-4, 7-13, 16-18, 20, 22, 24, and 26 are primarily for comparative purposes only.

## EXAMPLES

Example 1

A test was conducted to determine how much corn fiber oil could be extracted from corn bran by passing the corn bran through a flaking roll device. The test was initiated by obtaining an amount of corn bran, also known as corn fiber. The corn bran had an amount of moisture equal to 11.64% by weight of the total corn bran and a density equal to 10.8 lbs./$ft^3$.

Once the corn bran was obtained, approximately 1200 pounds of the corn bran was conditioned, by placing the corn bran in a French Stack Cooker, model number 15016, manufactured by the French Oil Mill company. The cooker had a

diameter of approximately 30 inches and contained four heated (4) horizontal trays. The corn bran was placed on the top tray in the cooker, with the corn bran being moved downward so that the corn bran contacted each of the four (4) trays, thereby exposing the corn bran to different temperatures on each of the trays. The corn bran material was evenly dispersed on the four (4) trays, with the corn bran material exposed to the trays heated with 100 psig of steam. Tray 1, which was the tray in the top portion of the cooker, had a temperature of about 115° F; tray 2, which was second from the top, had a temperature of about 96° F; tray 3, third from the top, had a temperature of about 94° F; and, the bottom tray, tray 4, had a temperature of about 184° F. The corn bran was left in the cooker for a period of time equal to about 20 minutes. Placement of the corn bran in the cooker resulted in a conditioned corn bran. The conditioned corn bran was then flaked by passing the corn bran into a flaking roll.

A flaking roll machine known as a Bauer Flaking Roll was used to flake the conditioned corn bran to form a densified flaked corn bran. The Bauer Flaking Roll was comprised of a pair of rollers each having a diameter of 14 inches and a width of 24 inches, with a gap setting between the two rollers equal to about .015 inches. One of the rollers rotated at a speed equal to about 377 revolutions per minute (rpms) and the other roller rotated at a speed equal to about 424 rpms. There was a 11% speed differential between the rolls. Passage through the flaking roll device formed the corn bran into a flaked corn bran.

The flaked corn bran was then placed in a Crown Model 2 extractor, which was a continuous extractor, which slowly pulled the corn bran through a hexane solvent bath. The densified corn bran was exposed to an amount of hexane so as to separate the corn fiber oil from the corn bran. As the corn bran passed through the extractor an amount of hexane solvent was mixed with the corn bran. The extractor had a capacity of 7.01 $ft^3$ so that an amount of corn bran equal to 142 lbs./hr. could pass through the extractor. Approximately 1137 lbs. of corn bran passed through the extractor, with the corn bran exposed to the hexane solvent for approximately 45 minutes. As such, it took approximately eight (8) hours to pass all of the corn bran through the extractor. Approximately 1600 lbs. of hexane was

added to the corn bran so that there was a solvent flow of 204 lbs./hour, the hexane was added in an amount sufficient to equal 1.4 parts by weight of solvent to 1 part by weight of corn bran. The temperature in the extractor averaged 124° F. After the hexane extraction of the corn bran was complete, approximately 1042 lbs. of corn bran remained, with the loss in weight primarily attributed to oil removal, moisture loss, and some of the corn bran remaining in the extractor.

The hexane, after contacting the corn bran, contained an amount of corn fiber oil. The corn fiber oil was then separated from the hexane by vaporizing the hexane using indirect steam at a temperature of approximately 220° F and a vacuum at approximately 12 inches of mercury. The hexane was vaporized in a Luwa thin film evaporator, fabrication number L-324. It was found that the hexane had removed approximately 20.8 lbs. of corn fiber oil, as that was the amount of corn fiber oil remaining in the batch tank after the hexane had been vaporized. This means that approximately 1.83% by weight of the corn fiber oil had been recovered from the corn bran.

Example 2

A test was conducted to determine how much corn fiber oil could be extracted from corn bran that had been passed through a flaking roll device and ground in a pin mill. The test was initiated by obtaining an amount of corn bran, also known as corn fiber. The corn bran had an amount of moisture equal to 11.64% by weight of the total corn bran and a density equal to about 10.8 lbs./ft$^3$.

Once the corn bran was obtained, approximately 1200 pounds of the corn bran was conditioned by placing the corn bran in a French Stack Cooker, model number 15016, manufactured by the French Oil Mill company. The French Stack Cooker had a diameter of approximately 30 inches and contained four (4) trays. When the corn bran was placed in the French Stack Cooker the corn bran moved downward so that the corn bran contacted each of the four (4) trays, thereby exposing the corn bran to different temperatures on each of the trays. The corn bran material was evenly dispersed on the four (4) trays, with the corn bran material exposed to trays heated with 100 psig of steam. Tray 1, which was the tray in the

top portion of the French Cooker, had a temperature of about 237° F; tray 2, which was second from the top, had a temperature of about 193° F; tray 3, third from the top, had a temperature of about 146° F; and, the bottom tray, tray 4, had a temperature of about 200° F. The corn bran was left in the French Cooker for a period of time equal to about 20 minutes. The cooking step resulted in a conditioned corn bran.

The conditioned corn bran was then flaked, with the same equipment, under the same conditions as disclosed in Example 1.

The flattened corn bran was then placed in pin mill grinder made by Alpine Contraplex Pin Mills, model # A-50 CW. The pin mill had two counter rotating sets of pins, which turned at 270 rpms. The flaked corn bran was ground into a flour, thereby forming a corn bran flour.

The corn bran flour was then placed in a Crown Model 4 extractor, which was a continuous extractor having a capacity of 1.94 $ft^3$, so that 63 lbs./hour of corn bran could pass through the extractor. As the corn bran flour passed through the extractor an amount of hexane solvent was mixed with the corn bran flour. Approximately 259 lbs. of the corn bran flour passed through the extractor, with the corn bran flour exposed to the hexane solvent in the extractor for approximately 45 minutes. The total extraction time for extracting 259 lbs. of densified corn bran flour took approximately four (4) hours. Approximately 260 lbs. of hexane was added to the corn bran flour so that there was a solvent flow of 66 lbs./hour and the hexane was added in an amount sufficient to equal 1 part by weight of solvent to 1 part by weight of corn bran flour. The temperature in the extractor averaged 114° F. After the hexane extraction of the corn bran flour was completed, approximately 180 lbs. of corn bran flour remained, with the loss in weight attributed to oil removal and moisture loss, as well as, some of the corn bran flour remaining in the extractor.

The hexane, after contacting the corn bran flour contained an amount of corn fiber oil. The corn fiber oil was then separated from the hexane by vaporizing the hexane using indirect steam at a temperature of approximately 220° F and a vacuum of approximately 12 inches of mercury. The hexane was vaporized in the same device disclosed in Example 1. It was found that the hexane had removed

approximately 6.5 lbs. of corn fiber oil, as that was the amount of the corn fiber oil remaining in the batch tank after the hexane had been vaporized. This means that approximately 2.51% by weight of the oil had been recovered from the corn bran.

Example 3

A test similar to Example 2 was conducted with the conditioning and flaking steps being the same as in Example 2. The corn bran had the same initial characteristics as that in Example 2 because it came from thc same batch as the corn bran used in Example 2. After the corn bran of Example 2 was flaked, about 300 lbs. was placed in a hammermill grinder, which had four (4) sets of five (5) hammers each rotating at 4500 rpms, with the hammer mill grinding the corn bran into a coarse densified corn bran. The screen size in the hammer mill was a U.S. mesh 1/8 inch screen. The coarse corn bran was then extracted under the same conditions recited in Example 1.

The amount of coarse corn bran placed in the extractor was 289 lbs. After extraction, the hexane contained an amount of corn fiber oil. The corn fiber oil was then separated from the hexane under the same conditions disclosed in Example 1. The hexane extraction resulted in the removal of approximately 6 lbs. of corn fiber oil, as that was the amount of oil remaining after the hexane had been vaporized. This means that approximately 2.08% by weight of the oil had been recovered from the corn bran.

Example 4

A test similar to Example 3 was conducted except the corn bran material was not flaked. The conditions and equipment were the same as Example 3 except for the exclusion of the flaking step, so that the corn bran was conditioned, ground in a hammer mill, and extracted. The corn bran came from the corn bran conditioned in Example 2 so that about 300 lbs. was ground in the hammer mill, the same as Example 3, and extracted. As such, 259 lbs. of milled corn bran was exposed to the hexane extraction step. The hexane was then separated from the corn fiber oil under the same conditions disclosed in Example 1, so that 1.1 lbs. of corn

fiber oil remained. This means that approximately 0.42% by weight of the oil had been recovered from the corn bran.

Example 5

An amount of corn bran, about 950 lbs., was passed through the same flaking roll device and under the same conditions disclosed in Example 1. The corn bran had an amount of moisture equal to about 11.64% by weight of the total corn bran and a density equal to about 10.8 lbs./ft$^3$. Once the corn bran had passed through the flaking roll device, the flaked corn bran was passed through the flaking roll device a second time, again under the same conditions as disclosed in Example 1. After passing the corn bran through the flaking roll a second time, the corn bran was ready for extraction.

The corn bran was then placed in a Crown Model 2 extractor, which was a continuous extractor having a capacity of 7.01 ft$^3$. The rate at which the corn bran was passed through the extractor was 101 lbs./hr. Approximately, 924 lbs. of corn bran passed through the extractor, with the corn bran exposed to a hexane solvent for approximately 90 minutes. The entire extraction process took a little over nine (9) hours. Approximately 1400 lbs. of a hexane solvent was added to the corn bran so that there was a solvent flow of 150 lbs./hour and the hexane was added in an amount sufficient to equal 1.5 parts by weight of solvent to 1 part by weight of corn bran. The temperature in the extractor averaged 124° F. After the hexane extraction of the corn bran was complete, approximately 834 lbs. of corn bran remained, with the loss in weight attributed to the oil removal, moisture loss, and some of the corn bran remaining in the extractor unit.

The hexane, after contacting the corn bran, contained an amount of corn fiber oil. The corn fiber oil was separated from the hexane the same as disclosed in Example 1. It was found that the hexane had removed approximately 24.9 lbs. of corn fiber oil from the corn bran, as that was the amount of oil remaining after the hexane had been vaporized. This means that approximately 2.69% by weight of the oil had been recovered from the corn bran.

Example 6

The same procedure was followed as disclosed in Example 5, except instead of exposing the densified corn bran to the hexane extraction step for 90 minutes, it was exposed to the hexane extraction step for 45 minutes. The amount of corn bran used was approximately 950 lbs. and the amount of corn bran that passed through the hexane extraction step was approximately 925 lbs. The rate at which the corn bran passed through the extractor was 202 lbs./hr. The solvent flow was 312 lbs./hr. It was observed that the shortened extractor time did not influence the extraction of the corn fiber oil and that approximately 2.69% by weight of the corn fiber oil was recovered.

Example 7

An amount of corn bran, 17.5 lbs., was dried for two (2) hours at 180° F by forcing heated air over the corn bran. The corn bran was dried in a Proctor & Schwartz tray type, forced draft oven, with Proctor & Schwartz located in Horsham, Pennsylvania. The drying lowered the moisture level in the corn bran from about 15% by weight of the corn bran to an amount ranging between about 2.0% and about 4.0% by weight of the corn bran. The dried corn bran was then ground by twice passing the corn bran through a Fitzpatrick mill, model #D, made by the Fitzpatrick company Elmhurst, Illinois. The Fitzpatrick mill is a screen type mill and has a plurality of hammers, which forced the corn bran through the screen. The screen had a size equal to a 1/8 inch perforation and the corn bran had different particle sizes so that on a #12 U.S. standard testing sieve 0.1% of the corn bran particles remained. On a #20 U.S. standard testing sieve 29.5% of the corn bran particles remained. On a #25 U.S. standard testing sieve 10.5% of the corn bran particles remained. On a #35 U.S. standard testing sieve 24.0% of the corn bran particles remained, and pan made up the remainder of the corn bran particles after passing through the hammer mill or Fitzpatrick mill.

After milling, the corn bran was subjected to three (3) liquid hexane washes, with the hexane covering the corn bran. The amount of hexane used to wash the corn bran was equal to about 56.5 lbs. The first hexane wash soaked the corn bran in hexane for five (5) hours. The second hexane wash soaked the corn bran in hexane for 15 hours and the third hexane wash soaked the corn bran in hexane for five (5) hours. The hexane extracted an amount of corn fiber oil and formed a miscella, which is a composition comprised of corn fiber oil and hexane. The miscella from the three hexane washes were combined. The corn fiber oil was then recovered from the hexane by vaporizing the hexane at a temperature of 160° F and a vacuum of 10 inches of mercury. The hexane was vaporized in a pilot plant rising film glass evaporator glass plant, made by Corning Glass Works, Corning, New York, and having serial #9015. After separation in the pilot plant evaporator, the miscella was placed in a rotavapor device having a temperature of 160° F and a vacuum of 25 inches of mercury. The rotavapor was used to separate the remaining hexane from the corn fiber oil. The rotavapor was model #R114, made by Buchi, and distributed by Beckman Instruments Inc, located in Westbury, New York. It was found that an amount of corn fiber oil equal to approximately 1.60% by weight of corn bran had been recovered. The remaining corn fiber oil was determined by weighing it on a standard scale.

Example 8

An amount of corn bran, 12.3 lbs., was twice ground in a Fitzpatrick mill the same as in Example 7. The corn bran had a moisture content equal to about 14% by weight of the corn bran. After grinding twice, the ground corn bran was then washed with hexane, the same as Example 7, and the corn fiber oil was recovered the same as disclosed in Example 7. An amount of corn fiber oil equal to about 0.632% by weight of the corn bran was recovered.

Example 9

A bucket of dried corn bran, about 17 lbs., which had been dried the same as Example 7, and which had a moisture level equal to about 3% by weight of the corn bran was ground in a contraplex pin mill. The contraplex had rotating elements on the door and in the mill of the device, with the elements rotating in opposite directions. The contraplex was operated with just the mill side pins rotating at 9048 rpm, with the contraplex made by Alpine and having model #250CWS. After passing through the contraplex, the ground corn bran was then washed with hexane, the same as Example 7, and the corn fiber oil was recovered the same as disclosed in Example 7. An amount of corn fiber oil equal to about 4.15% by weight of the corn bran was recovered.

Example 10

A bucket of corn bran, about 17 lbs., which was not dried and which had a moisture level equal to about 14% by weight of the corn bran, was flaked, in a flaking roll device at 800 psi. The flaking roll device was made by Ferrell-Ross, a division of Bluffton Agri/Industrial Corp., located in Bluffton Indiana. The flaking roll device had split rolls with the rolls being 24 inches by 24 inches. A 10% differential was used to flake the corn bran. The gap setting was not determined on the rolls, but the rolls were brought together and then barely separated. The pressure of 800 psi relates to the pressure between the flaking rolls, which comprise the flaking roll device. The flaked corn bran was then washed with hexane, the same as Example 7, and the corn fiber oil was recovered the same as disclosed in Example 7. An amount of corn fiber oil equal to about 2.34% by weight of the corn bran was recovered.

Example 11

A bucket of corn bran that had been dried the same as Example 7, and which had a moisture level equal to about 3% by weight of the corn bran, was flaked in a flaking roll device at 800 psi, in the same equipment listed in Example 10. The corn bran was then washed with hexane the same as Example 7 and the corn fiber oil

was recovered the same as disclosed in Example 7. An amount of corn fiber oil equal to about 2.75% by weight of the corn bran was recovered.

Example 12

A bucket of corn bran which was not dried and which had a moisture level equal to about 19% by weight of the corn was flaked, in a flaking roll device at 800 psi in the same equipment listed in Example 10. After flaking, the corn bran was dried for two (2) hours at 180° F, in the same equipment listed in Example 7, so that the moisture level after drying was equal to about 2.5% by weight of the corn bran. After drying, the corn bran was subjected to a hexane extraction method in which a five (5) gram sample of the ground corn bran was analyzed in a Soxtec system HTG, manufactured by Foss North America Inc., Eden Prarie, Minnesota. The test determines how much corn fiber oil is found to be available for extraction in the corn bran. The test involved placing the five gram corn bran sample into a thimble and plugging the thimble with glass wool. The thimble was then placed in the Soxtec. Next, 40 ml of hexane was added to an extraction cup, with the extraction cup placed on top of a hot plate. The thimble was then placed in the extraction cup for 15 minutes. The thimble was then removed from the cup and rinsed with hexane for two hours, with the hexane passing through the thimble and into the extraction cup. The hexane was then boiled out of the cup, with the boiling off of the hexane taking about 15 minutes. The cup was then placed in an oven heated to 130° C for 30 minutes. The cup was cooled in a desiccator for 20 minutes and weighed. The cup had been weighed prior to the addition of the corn bran. The amount of available corn fiber oil was determined by the different weights of the cup before and after extraction. An amount of corn fiber oil equal to about 2.71% by weight of the corn bran was found to be available, meaning this amount of corn fiber oil could be extracted using standard hexane extraction methods.

Example 13

A bucket of corn bran which was not dried and which had a moisture level equal to about 19% by weight of the corn was flaked, in a flaking roll device at

800 psi in the same equipment listed in Example 10. After flaking, the corn bran was dried for two (2) hours at 180° F, in the same equipment listed in Example 7, so that the moisture level after drying was equal to about 2.5% by weight of the corn bran. The corn bran was then extracted with hexane, the same as Example 12. An amount of corn fiber oil equal to about 2.61% by weight of the corn bran was found to be available.

Example 14

A bucket of corn bran having a moisture level equal to about 19% by weight of the corn bran was flaked twice, in a flaking roll device at 800 psi in the same equipment listed in Example 10. After double flaking, the corn bran was dried for two (2) hours at 180° F, in the same equipment listed in Example 7, so that the moisture level after drying was equal to about 2.5% by weight of the corn bran. The corn bran was then extracted with hexane, the same as Example 12. An amount of corn fiber oil equal to about 3.31% by weight of the corn bran was found to be available.

Example 15

A bucket of corn bran having a moisture level equal to about 19% by weight of the corn bran was flaked twice, in a flaking roll device at 800 psi in the same equipment listed in Example 10. After double flaking, the corn bran was dried for two (2) hours at 180° F, in the same equipment listed in Example 7, so that the moisture level after drying was equal to about 2.5% by weight of the corn bran. The corn bran was then extracted with hexane, the same as Example 12. An amount of corn fiber oil equal to about 3.27% by weight of the corn bran was found to be available.

Example 16

A bucket of corn bran having a moisture level equal to about 19% by weight of the corn bran was flaked, in a flaking roll device at 800 psi in the same

equipment listed in Example 10. After flaking, the corn bran was dried for two (2) hours at 180° F, in the same equipment listed in Example 7, so that the moisture level after drying was equal to about 2.5% by weight of the corn bran. The corn bran was then extracted with hexane, the same as Example 12. An amount of corn fiber oil equal to about 2.45% by weight of the corn bran was found to be available.

Example 17

A bucket of corn bran having a moisture level equal to about 19% by weight of the corn bran was flaked, in a flaking roll device at 800 psi in the same equipment listed in Example 10. After flaking, the corn bran was dried for two (2) hours at 180° F, in the same equipment listed in Example 7, so that the moisture level after drying was equal to about 2.0% by weight of the corn bran. The corn bran was then extracted with hexane, the same as Example 12. An amount of corn fiber oil equal to about 2.67% by weight of the corn bran was found to be available.

Example 18

A bucket of corn bran having a moisture level equal to about 18.1% by weight of the corn bran was flaked, in a flaking roll device at 800 psi in the same equipment listed in Example 10. After flaking the corn bran was dried for two (2) hours at 180° F, in the same equipment listed in Example 7, so that the moisture level after drying was equal to about 2.5% by weight of the corn bran. The corn bran was then extracted with hexane, the same as Example 12. An amount of corn fiber oil equal to about 2.74% by weight of the corn bran was found to be available.

Example 19

A bucket of corn bran having a moisture level equal to about 18.1% by weight of the corn bran was flaked twice, in a flaking roll device at 800 psi in the same equipment listed in Example 10. After flaking twice, the corn bran was dried for two (2) hours at 180° F, in the same equipment listed in Example 7, so that the moisture level after drying was equal to about 2.5% by weight of the corn bran. The corn bran was then extracted with hexane, the same as Example 12. An amount of

corn fiber oil equal to about 3.31% by weight of the corn bran was found to be available.

Example 20

A bucket of corn bran having a moisture level equal to about 18.1% by weight of the corn bran was flaked, in a flaking roll device at 800 psi in the same equipment listed in Example 10. After flaking, the corn bran was dried for two (2) hours at 180° F, in the same equipment listed in Example 7, so that the moisture level after drying was equal to about 2.5% by weight of the corn bran. The corn bran was then extracted with hexane, the same as Example 12. An amount of corn fiber oil equal to about 2.65% by weight of the corn bran was found to be available.

Example 21

A bucket of corn bran having a moisture level equal to about 18.1% by weight of the corn bran was flaked twice, in a flaking roll device at 800 psi in the same equipment listed in Example 10. After double flaking, the corn bran was dried for two (2) hours at 180° F, in the same equipment listed in Example 7, so that the moisture level after drying was equal to about 2.5% by weight of the corn bran. The corn bran was then extracted with hexane, the same as Example 12. An amount of corn fiber oil equal to about 3.24% by weight of the corn bran was found to be available.

Example 22

A bucket of corn bran having a moisture level equal to about 18.1% by weight of the corn bran was flaked, in a flaking roll device at 800 psi in the same equipment listed in Example 10. After flaking, the corn bran was dried for two (2) hours at 180° F, in the same equipment listed in Example 7, so that the moisture level after drying was equal to about 2.5% by weight of the corn bran. The corn bran was then extracted with hexane, the same as Example 12. An amount of corn fiber oil equal to about 2.78% by weight of the corn bran was found to be available.

Example 23

A bucket of corn bran having a moisture level equal to about 18.1% by weight of the corn bran flaked twice, in a flaking roll device at 800 psi in the same equipment listed in Example 10. After flaking twice, the corn bran was then dried for two (2) hours at 180° F, in the same equipment listed in Example 7, so that the moisture level after drying was equal to about 2.5% by weight of the corn bran. The corn bran was then extracted with hexane, the same as in Example 12. An amount of corn fiber oil equal to about 3.22% by weight of the corn bran was found to be available.

Example 24

An amount of corn bran, 20 lbs., having a moisture level of 17.8% by weight was flaked in a flaking roll device at 700 psi in the same equipment listed in Example 10. The corn bran was then extracted with hexane, the same as Example 12. An amount of corn fiber oil equal to about 2.60% by weight of the corn bran was found to be available.

Example 25

An amount of corn bran, 20 lbs., having a moisture level of 17.8% by weight was flaked twice in a flaking roll device at 700 psi in the same equipment listed in Example 10. The corn bran was then extracted with hexane, the same as Example 12. An amount of corn fiber oil equal to about 2.97% by weight of the corn bran was found to be available.

Example 26

An amount of corn bran, 20 lbs., having a moisture level of 17.8% by weight was flaked in a flaking roll device at 900 psi in the same equipment listed in Example 10. The corn bran was then extracted with hexane, the same as Example 12. An amount of corn fiber oil equal to about 2.90% by weight of the corn bran was found to be available.

Example 27

An amount of corn bran, 20 lbs., having a moisture level of 17.8% by weight was flaked twice in a flaking roll device at 900 psi in the same equipment listed in Example 10. The corn bran was then extracted with hexane, the same as Example 12. An amount of corn fiber oil equal to about 3.15% by weight of the corn bran was found to be available.

Example 28

An amount of corn bran, 20 lbs., having a moisture level of 16.0% by weight was flaked twice in a flaking roll device at 900 psi in the same equipment listed in Example 10. After flaking the corn bran was dried for two (2) hours at 180° F in the same equipment listed in Example 7. The corn bran was then extracted with hexane, the same as Example 12. An amount of corn fiber oil equal to about 3.95% by weight of the corn bran was found to be available.

Example 29

An amount of corn bran, 20 lbs., having a moisture level of 16.0% by weight was flaked twice in a flaking roll device at 700 psi in the same equipment listed in Example 10. After flaking the corn bran was dried for two (2) hours at 180° F in the same equipment listed in Example 7. The corn bran was then extracted with hexane, the same as Example 12. An amount of corn fiber oil equal to about 3.82% by weight of the corn bran was found to be available.

As can be seen from the above examples, the most corn fiber oil was extracted when the corn bran was flaked twice. In particular, Examples 5, 6, 14, 15, 19, 21, 23, 25, and 27 showed superior extraction results as compared to other methods. Examples 28 and 29 showed the best results, with the corn bran being flaked twice and then dried. Also, flaking alone and flaking plus an additional step showed better extraction results than simply grinding the corn bran and extracting the corn fiber oil from the corn bran. As can be seen, Examples 4, 7, and 8 did not include a corn bran flaking step and consequently the resulting amount of corn fiber

oil extracted was less than the methods involving flaking the corn bran at least once and far less than the methods involving double flaking. Also, it appeared that dried corn bran allowed for greater extraction than wet corn bran.

If the corn bran is flaked only once then it is preferred, but not required, to further treat the corn bran. Such treatment can include grinding the corn bran in a variety of ways, including using a pin mill, a hammer mill, or any other type of device that will reduce the particle size of the corn bran.

Thus, there has been shown and described a novel method and composition related to removing corn fiber oil from corn bran which fulfill all the objects and advantages sought therefore. It is apparent to those skilled in the art, however, that many changes, variations, modifications, and other uses and applications for the subject method and composition are possible, and also such changes, variations, modifications, and other uses and applications which do not depart from the spirit and scope of the invention are deemed to be covered by the invention which is limited only by the claims which follow.

What is claimed is:

1. A method for extracting corn fiber oil from corn bran, wherein said method comprises the steps of:

(a) flaking an amount of the corn bran by passing the corn bran through a flaking roll device so as to form an amount of flaked corn bran;

(b) passing said flaked corn bran through a flaking roll device to form an amount of twice flaked corn bran; and,

(c) exposing said twice flaked corn bran to an amount of solvent so as to extract said corn fiber oil from said twice flaked corn bran thereby forming a miscella, with said miscella containing an amount of said corn fiber oil equal at least about 2% by weight of the corn bran.

2. The method of claim 1 wherein said flaking roll device in step (a) has a pair of flaking rolls having a gap setting ranging between about 0 and about 0.015 inches and a differential ranging between about 1% and about 20%.

3. The method of claim 1 wherein said flaking roll device in step (b) has a pair of flaking rolls having a gap setting ranging between about 0 and about 0.015 inches and a differential ranging between about 1% and about 20%.

4. The method of claim 1 wherein said method further includes a corn bran drying step, whereby the corn bran is dried prior to exposing said twice flaked corn bran to the solvent so that said twice flaked corn bran has a moisture content equal to no more than about 15% by weight of said twice flaked corn bran.

5. The method of claim 1 wherein said method includes a step whereby the solvent is vaporized away from said miscella so that said corn fiber oil remains.

6. The method of claim 1 wherein said method more preferably removes an amount of said corn fiber oil equal to at least about 3% by weight of the corn bran.

7. The method of claim 1 wherein the said method allows for removal of at least about 40% by weight of said total corn fiber oil.

8. The method of claim 4 wherein said twice flaked corn bran preferably has a moisture content ranging between about 2% and about 8% by weight of said twice flaked corn bran.

9. The method of claim 1 wherein the solvent is a non-polar solvent.

10. The method of claim 9 wherein said non-polar solvent is selected from the group consisting of hexane, supercritical $CO_2$, butane, isooctane, propane, acetone, isohexane, heptane, and methylpentane.

11. A method for preparing corn bran for solvent extraction of corn fiber oil from the corn bran, wherein said method comprises the steps of:

(a) passing the corn bran through a flaking roll so as to form an amount of flaked corn bran; and,

(b) passing said flaked corn bran through a flaking roll to form an amount of twice flaked corn bran, whereby an amount of said corn fiber oil in said twice flaked corn bran can be extracted by a solvent.

12. The method of claim 11 wherein said flaking roll in step (a) has a gap setting ranging between about 0 inches and about 0.015 inches and a differential ranging between about 1% and about 20%.

13. The method of claim 11 wherein said flaking roll in step (b) has a gap setting ranging between about 0 inches and about 0.015 inches and a differential ranging between about 1% and about 20%.

14. The method of claim 11 wherein said method allows for at least about 2% by weight of the corn fiber oil to be extracted.

15. The method of claim 11 wherein said method includes a step whereby the corn bran is dried so that the corn bran has a moisture level equal to no more than about 15% by weight of the corn bran.

16. A method for preparing corn bran for solvent extraction of corn fiber oil from the corn bran, wherein said method includes the step of passing the corn bran through a flaking roll device at least once to form a flattened corn bran that permits an amount of corn fiber oil to be extracted from said flattened corn bran.

17. The method of claim 16 wherein said method includes a step whereby said flattened corn bran is passed into a hammer mill to be ground to a smaller particle size.

18. The method of claim 16 wherein said method includes a step whereby said flattened corn bran is passed into a pin mill to be ground to a smaller particle size.

19. The method of claim 16 wherein said method includes a step whereby said flattened corn bran is ground to a smaller particle size.

20. A treated corn bran that allows for ready extraction of corn fiber oil from said treated corn bran, wherein said treated corn bran has a moisture level equal to no more than 15% by weight of said treated corn bran, a

thickness ranging between about 0.005 inches and about 0.015 inches, and an average particle size ranging between about a #10 U.S. standard testing sieve and a #60 U.S. standard testing sieve.

## INTERNATIONAL SEARCH REPORT

International application No.
PCT/US99/07778

**A. CLASSIFICATION OF SUBJECT MATTER**

IPC(6) :A23D 7/00, 9/00; A23L 1/28; C07C 11/00; C11B 1/10
US CL :426/ 417, 425, 429, 601; 554/8, 9, 12
According to International Patent Classification (IPC) or to both national classification and IPC

**B. FIELDS SEARCHED**

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 426/ 417, 425, 429, 601; 554/8, 9, 12

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

APS, CAS ONLINE, FSTA, AGRICULTURE

**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

| Category* | Citation of document, with indication, where appropriate, of the relevant passages | Relevant to claim No. |
|---|---|---|
| P,E | SNYDER et al. Concentration of Phytoseterols for Analysis by supercritical fluid extraction. Journal of the American Oil Chemist. June 1999, Vol. 76, No. 5, pages 717-721. | 1-20 |

☐ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

* Special categories of cited documents:

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Date of mailing of the international search report

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